

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/576,033
Applicant : Milliren, Charles M.
Filed : April 14, 2006
Title : VISCOELASTIC FOAM LAYER AND COMPOSITION
Examiner : John M. Cooney
Docket No. : 36211US1
Customer No. : 000116

DECLARATION OF CHARLES M. MILLIREN, Ph.D.

Charles M. Milliren, Ph.D., having knowledge of the facts set forth herein, declares as follows:

1. I am the named inventor of the present patent application.
2. I presently reside at 12589 Harold Drive, Chesterland, OH 44026.
3. I received a Bachelor of Science in Chemistry from Ursinus College, Collegeville, PA in 1969. I received a Ph.D. in Organic Chemistry from the University of North Carolina at Chapel Hill, North Carolina in 1975.
4. Following my formal education, I was employed by Mobay Chemical Corporation/Miles Inc., Pittsburgh, Pennsylvania, as a research scientist from 1976 to 1991. Subsequently I worked for Bayer Corporation USA in Pittsburgh, Pennsylvania, also as a research scientist, from 1991 to 2001. From 2002 till the present I have been employed by Team Wendy, LLC (f/k/a Coit Road Incubator), Cleveland Ohio and engaged primarily in the research, development and testing of foams for energy absorption applications. Team Wendy, LLC is a related company to Intellectual Property Holdings, LLC, the assignee of the present application.
5. I am an expert in foam chemistry, particularly polyurethane foam chemistry and morphology, with over 30 years of professional research and

development experience. I have authored or co-authored several publications in the field of polyurethane foam chemistry and morphology, a partial list of which is attached hereto as Attachment A.

6. I also am an inventor or co-inventor of numerous U.S. patents relating to polyurethane foam chemistry and morphology, which are listed in Attachment B.

7. I have reviewed the Office action dated December 17, 2008.

8. The specific chemical components that go into a polyurethane foam are outcome-determinative for the physical and behavioral characteristics for the resulting foam. In addition to polyol(s), polyurethane foam formulations may include blowing agent(s), catalyst(s), chain extender(s), crosslinker(s), pigment(s), surface active agent(s), filler(s), flame retardant(s), etc., and polyisocyanate(s). Each component can affect the characteristics of the final foam in an unpredictable way based on its effect on the foam's morphology and physico-chemical structure, which cannot be specified a priori. In general, the polyols used in polyurethane foams can be polyether or polyester types. For polyether foams the initiator molecule and the alkyleneoxides (e.g. ethylene oxide [EO] and polyethylene oxide [PO]) used for chain extension are extremely important in determining the solubility, compatibility, reactivity, surface tension, stability and function of the reacting polyol. The alkyleneoxides (EO, PO) make an important contribution toward determining the physical characteristics of the resulting polyurethane foam, i.e., the foam's morphology, which is largely determined by the molecular weight, structure and reactivity of the polyol(s) in addition to other factors. The morphology determines the type and amount of important physical interactions in the resulting foam, which contribute to the foam's rigidity.

9. Ethylene oxide extension units in a polyol tend to increase the flexibility (decrease the rigidity) of a polyurethane foam because EO extension produces a regular linear structure with a higher number of degrees of freedom than does extension by propylene oxide (PO). Conversely, propylene oxide extension units tend to decrease foam flexibility (increase rigidity) because of a reduced number of degrees of freedom. This is due to the pendant methyl groups present in PO-extended polyols, which are not

present in EO extension units or in exclusively EO-extended polyols. Therefore, a polyurethane foam made from a substantial proportion of amine-based polyols (which are typically used to produce rigid foams), that are propylene-oxide extended and have substantially no ethylene oxide extension units, would be expected to be quite a rigid foam, having a much reduced degree of flexibility. Such a foam certainly would not be expected to exhibit a flexible recovery following a deflection.

10. However, very surprisingly, it has been found that substantially fully-recoverable semi-rigid viscoelastic foams can be prepared using in the 'Part B' composition for making polyurethane foam a substantial proportion (at least 10 parts by weight out of 100 parts by weight total polyols) of amine-based polyether polyols that are propylene-oxide extended and have substantially no ethylene oxide extension units.

11. Regarding the references cited by the examiner, none of them discloses or even remotely suggests a semi-rigid, recoverable viscoelastic foam. Certainly none of them discloses achieving such a polyurethane foam using the substantial proportion of amine-based polyether polyol recited in the independent claims of the present application, which are propylene-oxide extended and have substantially no ethylene oxide extension units.

12. First, the foams in Apichatachutapan are soft foams, not semi-rigid foams, as evidenced by the data presented in the Examples of Apichatachutapan. Specifically, the foams in this reference have been tested for physical properties characteristic of soft flexible foams (25% and 65% IFD, sag factor) and the resulting data are typical of such foams; see Table 2 in Apichatachutapan. Conversely, the "semi-rigid" foams as claimed in the present application could not be tested for IFD or sag at least because they are too rigid to fall within the property limits of soft flexible foams. On the other hand, the present application presents extensive data for semi-rigid foams showing energy absorption characteristics at various dynamic impact speeds. Dynamic impact testing is totally unsuitable for flexible foams as in Apichatachutapan because such foams do not exhibit any appreciable degree of dynamic impact energy attenuation and would be instantaneously completely compressed (they would bottom out) with no

energy absorption to speak of. Apichatachutapan is concerned primarily with providing a high density flexible foam that exhibits flame retardant properties without the incorporation of any separate flame retardant additive (see para. [0002] and rest of application). No mention or hint of energy absorption properties, particularly against a dynamic impact, (and hence no suggestion to prepare a semi-rigid foam) is to be found in Apichatachutapan.

13. Regarding Lutter, the foams in this reference are soft, flexible foams (see col. 4, line 45 and col. 8, line 50) which are completely different from the semi-rigid foams described in the present application. Lutter does not disclose or discuss dynamic impact properties for the foams therein, because soft, flexible foams are unsuited for this purpose. Again, no reason to prepare a foam having energy-absorptive properties is evident.

14. Likewise, Falke describes only flexible polyurethane foams (col. 1, line 5) designed primarily as sound absorbers in carpet backing (col. 1 and col. 2) with a loss factor (col., line 40) greater than 0.3. Falke does not discuss or show any dynamic impact properties for the disclosed flexible foams, principally because such foams are totally unsatisfactory for dynamic impact attenuation.

15. Conversely, the semi-rigid viscoelastic foams now claimed are directly suited for dynamic impact attenuation applications as evidenced by the substantial dynamic impact test data provided in the specification. There would be no reason to make, or even to expect one could make, a semi-rigid foam falling within the scope of the present claims based on the teachings of any of the cited references, in particular because all of them disclose flexible foams and none of them even hints at a semi-rigid foam or at any application, such as dynamic impact energy attenuation, that calls for a semi-rigid foam.

16. The present invention is based on the extremely surprising result that the combination of a substantial amount of an amine-based polyether polyol, or a mixture of such polyols, that is/are propylene oxide extended, together with at least one other filled and/or unfilled polyether polyol and an appropriate amount of isocyanate, produces a

semi-rigid viscoelastic foam that has proven very effective to attenuate impact force across a very wide range of dynamic impact speeds; e.g. ranging from 2-6 meters per second, or broader, and is also recoverable after impact. This result was particularly surprising because amine-based polyols are usually considered to be rigid foam precursors, and it was quite surprising that the use of such polyols, in relatively significant amounts with other polyols, and particularly having substantially no ethylene oxide extension units, would produce such effective semi-rigid foams that are so well suited to dynamic impact energy attenuation applications, yet still recover following an impact. Even more surprising was the breadth of impact speeds over which impact force could be effectively attenuated using these foams compared to a conventional rigid foam such as EPS, as seen in the Examples in the application and discussed below.

17. It was particularly surprising that amine-based polyols having substantially no ethylene oxide extension units would produce such an effective semi-rigid and recoverable viscoelastic polyurethane foam because EO extension is usually employed in semi-rigid foams to provide a requisite degree of flexibility compared to rigid, non-recoverable foams. In addition, EO extension helps contribute sufficient reactivity to the polyol to produce a stable foam morphology (structure) in the resultant semi-rigid foam, and produces excellent cure characteristics generally desired of semi-rigid foams. A recoverable, semi-rigid viscoelastic foam based on at least 10 parts by weight propylene oxide-extended amine-based polyol having substantially no ethylene oxide extension units was very surprising.

18. Example 1 in the application describes eight different foams all made according to compositions of propylene oxide extended amine-based polyols (having no ethylene oxide extension units) with filled and unfilled (trifunctional, glycerin based) polyols as claimed. Table 3 of that Example shows that all eight of these foams performed comparably to conventional expanded polystyrene (EPS), which is the conventional rigid foam used in bicycle helmets to attenuate high speed impacts, for an

impact velocity of nominally 6.23 meters/second¹. In fact, six of the eight foam formulations tested actually exhibited lower peak g-values (breakthrough acceleration values) than EPS. This was a very highly surprising and unexpected result for a semi-rigid viscoelastic foam that substantially recovered following each impact. Further surprising was the fact that mixtures of two and three amine-based polyether polyols in the compositions in Example 1 (Table 2), all of which contained substantially no ethylene oxide extension units, produced foams that nonetheless substantially recovered following impact as seen in the example.

19. Example 2 and Figs. 1-12 demonstrate further the ability of an exemplary semi-rigid viscoelastic foam according to the invention, Foam No. 2 from table 2, to adequately and effectively attenuate dynamic impacts of low (2 meters/second) to high (6 meters per second and beyond) compared to conventional EPS. As the data demonstrate, EPS is far less effective than the foams of the invention to attenuate low speed impacts because of its substantial rigidity. In addition, EPS, being a rigid foam, is irreversibly and destructively crushed on impact of sufficient force and thus did not recover and could not be reused. Conversely, the foams according to the invention, being semi-rigid and viscoelastic, were effective to attenuate a wide range of impact forces and did recover substantially, permitting their reuse for multiple impacts.

20. The combination of these widely differing yet highly desirable properties:

- semi-rigid foam exhibiting comparable performance to EPS following high-speed impact (6.23 m/s) using a rigid spherical or circular impactor under the conditions disclosed in the specification;
- viscoelastic foam that behaves comparably to a flexible foam for relatively low-speed impacts (e.g. 2 m/s) using the impactors and conditions disclosed in the specification;
- a foam that substantially recovers following high speed impact;

¹ Conventional EPS is the closest prior art to the present foams, which provide impact protection comparable to EPS for high-speed impacts at 6.23 m/s under the conditions described in the Examples. However, unlike EPS, the present foams are recoverable and can be reused, and also provide a measure of impact protection at lower speeds (e.g. 2 m/s). At such low speeds, impact force is substantially transmitted through EPS, thereby providing little or no impact protection at all.

- a foam that following recovery, exhibits negligible loss in strength and its ability to absorb another high-speed impact (6.23 m/s) using the impactors and conditions disclosed in the specification;

In a single foam that incorporates a substantial proportion (≥ 0 parts by weight, out of a total of 100 parts by weight) of amine-based polyether polyols that are propylene-oxide extended and have substantially no ethylene oxide extension units, was an incredibly surprising and unexpected result, which could not have been predicted *a priori* by a person having ordinary skill in the art who has had placed in front of him every conceivable species of isocyanate, polyol initiator, polyol extension unit, catalyst, filler, etc. that has ever been used or could be used to prepare any polyurethane foam, with no guidance how to combine them or in what proportions, or even what the properties of the ultimate foam should be.

21. A person having ordinary skill in the art certainly would not have expected to achieve a polyurethane foam having the properties recited above, using the proportion of amine-based, propylene-oxide extended polyol, having substantially no ethylene oxide extension units, in the Part B composition for a polyurethane foam as recited in claims 35 and 54 of the present application.

22. Nothing in the prior art references suggests the combination of an amine-based polyether polyol(s), which are very reactive and typically used for rigid foams, with the filled and/or unfilled polyols as claimed to produce a semi-rigid viscoelastic foam exhibiting the aforementioned properties. Furthermore, nothing in the prior art suggests the amine-based polyol(s) should be propylene oxide extended and have substantially no ethylene oxide extension units as in the claimed formulations. The combination of these components to produce the semi-rigid viscoelastic foam having the properties described above truly was a very surprising and unexpected result that could not have been predicted *a priori* based on selection of polyols and/or other reagents for the foam compositions. Certainly, there would have been no expectation of success, starting from any of the cited references, to produce a semi-rigid viscoelastic foam as claimed based significantly on propylene oxide extended amine-based polyols, at least because all those references are directed to flexible foams and no reason is

provided or evident in any of them why or how amine-based polyols can be used to provide an effective semi-rigid viscoelastic foam.

23. I have carefully and thoroughly reviewed the Apichatachutapan, Lutter and Falke references applied by the Examiner. As an expert in polyurethane chemistry with over thirty years of professional research experience in the field, the vast disclosures of literally tens of different species for each class of compound typically found or that can be incorporated in a polyurethane foam provides no special motivation or suggestion to select particular ones of each said class to combine them in any other way than the respective references actually did. In the references, there is no combination whatsoever incorporating the proportion and chemistry of the amine-based polyether polyols as presently claimed. And rightfully so, as these references all describe flexible foams. None of these references presents any reason or motivation to produce the formulation as now recited in either the independent claims 35 and 54 in the present application. Nor would any reason to select the particular compositions there claimed be evident to the skilled artisan in view of those references, at least because:

- a) one would not expect to obtain a recoverable semi-rigid viscoelastic foam using the proportion of propylene-oxide extended amine-based polyether polyols having substantially no ethylene oxide extension units as claimed; and
- b) the vast laundry lists of tens of species for each general class of components that can make up a polyurethane foam, as found in the references, do not suggest to the skilled artisan any particular foam formulation, for any purpose, based on knowledge one could reasonably expect the ordinarily-skilled artisan to possess.

24. In addition, the reason proposed by the Examiner to suggest it would have been obvious to use amine-based polyols in any of the three cited references is incorrect. In connection with all three references, the Examiner stated in the Office action that it would have been obvious to substitute amine initiators (resulting in amine-based polyols) for the initiators actually used "for the purpose of providing their isocyanate reactive effect in the preparations of [the reference] in order to arrive at the products...of applicants' claims..." It is true that amine initiators result in highly reactive

polyols. This produces foams with high degrees of cross-linking compared to foams produced using polyols based on other initiators, e.g. glycerin. Higher cross-linking rates typically result in much stiffer, more rigid foams as known in the art. Therefore, the high reactivity of amine-based polyols cited by the Examiner is actually a reason not to use them in any of the references (certainly in the proportions claimed), which all clearly are directed to flexible or soft foams as noted above. In addition, the high reactivity of amine-based polyols also makes them a unlikely candidate to produce the semi-rigid viscoelastic foams that are now claimed, which are recoverable after deformation. As already explained, it was highly unexpected that a foam formulation containing the proportion of amine-based (reactive) polyols as required in the claims would yield a foam that is not only viscoelastic, but that substantially recovers following deformation and can be reused. A person of ordinary skill in the art would not expect this result based on using amine-based polyols in the claimed proportions (certainly not ones having substantially no ethylene oxide extension units), and therefore would have had no expectation of success to achieve the recoverable semi-rigid viscoelastic foam that is claimed.

25. Finally, although the references mention amine initiators, any person of ordinary skill in the art would recognize that all of them are directed to flexible foams, and that the mention of amine initiators and numerous other ingredients that might be used to make all kinds of polyurethane foams was mere laundry-listing of every potential ingredient the writer could think of, without any consideration whether or how to select any subset or combination of them to produce a particular foam. A person of ordinary skill in the art would not be led to use the amine-based polyols now claimed, particularly having substantially no ethylene oxide extension units as claimed, to make *either* any foam disclosed in Lutter, Falke or Apichutachatapan, *or* any foam of the present claims. To prepare the present recoverable, semi-rigid viscoelastic foams, I initially had all of these potential ingredients at my disposal, and I undertook exhaustive experimentation to develop a foam having the target properties. As noted above I have over thirty years of experience in research and development of polyurethane foams. I could not possibly have guessed at the outset that a foam composition made from a 'part B' composition, as known in the art, having at least 10 parts by weight (out of 100

parts by weight total polyol) propylene oxide-extended amine-based polyether polyol with substantially no ethylene oxide extension units, would produce the semi-rigid, viscoelastic and recoverable foam I had hoped to produce. That was a surprising and unexpected result based on my over thirty years of professional experience, which no person of ordinary skill in this art could have predicted *a priori*, or would have found obvious from the lists of potential ingredients in Lutter, Falke or Apichutachatapan.

26. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Charles M. Milliren, Ph.D.

12/17/2008
Date

ATTACHMENT A:
List of Relevant Publications

1. 33RD Polyurethanes Technical/Marketing Conference, Sept. 30-Oct. 3, 1990, pp 28-31, A New MDI Foam Technology for Seating and Headrest Applications.
2. *Plastics Engineering*, Jan. 1991, page 23, "A New Non-CFC, MDI Based Flexible Foam Technology."
3. Polyurethanes Expo 99, Sept. 12-15, 1999, pp. 151-161, "Low Compression Set – High Elongation: A Paradox?"
4. Paper by Orbseal LLC and Bayer Corporation, Oct. 1, 2001, New PU Cavity Filling Foam for Sound Abatement in Car Body Shells.

ATTACHMENT B:
List of U.S. Patents of which I am Inventor or Co-Inventor

1. 4,876,292 - Isocyanate reactive mixture and the use thereof in the manufacture of flexible polyurethane foams
2. 5,028,637 - Isocyanate reactive mixture and the use thereof in the manufacture of flexible polyurethane foams
3. 5,252,624 - System for the production of toluene diisocyanate based flexible foams and the flexible foams produced therefrom
4. 5,405,886 - System for the production of toluene diisocyanate based flexible foams and the flexible foams produced therefrom 5,415,802 Water Blown, Energy Absorbing Foams
5. 5,417,880 - Water blown, energy absorbing foams
6. 5,449,700 - Water blown, energy absorbing foams
7. 5,457,137 - Water blown, energy absorbing foams
8. 5,874,485 - Flexible foams and flexible molded foams based on allophanate-modified diphenylmethane diisocyanates and processes for the production of these foams
9. 6,235,138 - Polyurethane foam/PVC laminate for automotive instrument panels